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Report MAT-75-78

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NAVAL SHIP RESEARCH AND DEVELOPMENT CENTER

Bethesda, Md. 20084

AD B009649

ELASTOMERS FOR SERVICE AS SEALS FOR ENGINE LUBRICANTS AND HYDRAULIC FLUIDS

by
William Klemens and Paul Lagally

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RESEARCH AND DEVELOPMENT REPORT

Elastomers for Service as Seals for Engine Lubricants and Hydraulic Fluids

February 1976 Report MAT-75-78

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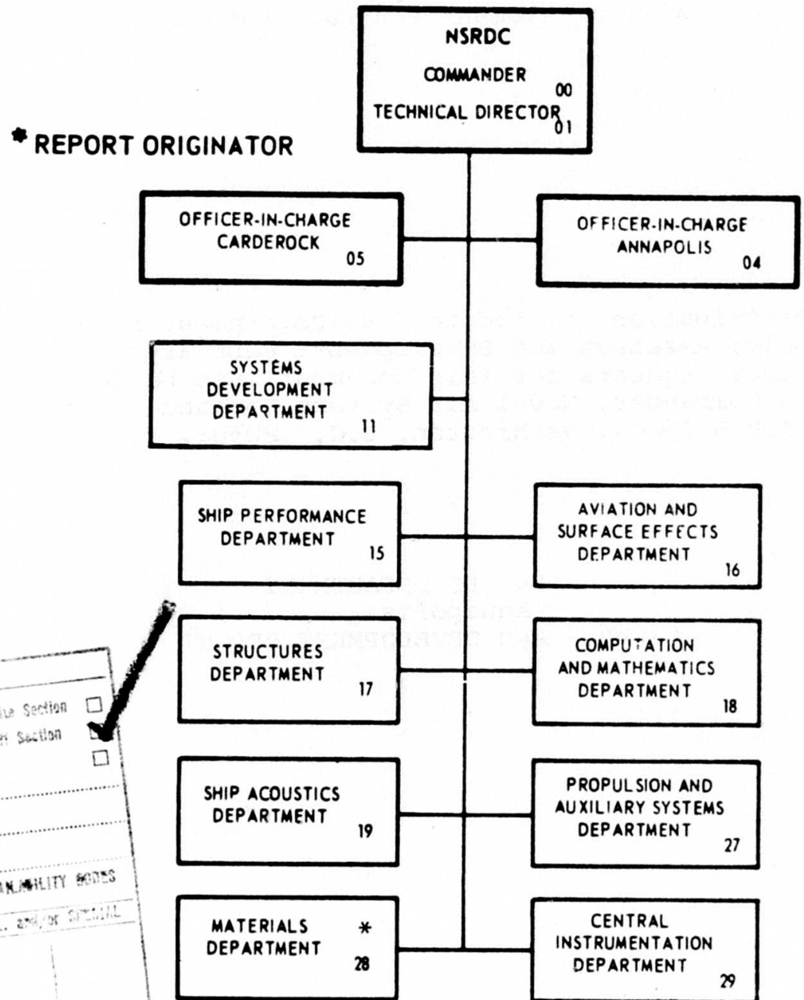

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REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER
14 MAT-75-78 ✓

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER
9

4. TITLE (and Subtitle)
6 ELASTOMERS FOR SERVICE AS SEALS FOR ENGINE LUBRICANTS AND HYDRAULIC FLUIDS,

5. TYPE OF REPORT & PERIOD COVERED
Research and Development, rept.,

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
William/Klemens and Paul/Lagally
10

8. CONTRACT OR GRANT NUMBER(s)
12 16p.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
David W. Taylor Naval Ship R&D Center
Annapolis, Maryland 21402 ✓

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
17 WF 54-544-205
Work Unit 2841-516

11. CONTROLLING OFFICE NAME AND ADDRESS
David W. Taylor Naval Ship R&D Center
Bethesda, Maryland 20084

12. REPORT DATE
11 Feb[redacted]976

13. NUMBER OF PAGES
11

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)
16 WF54-544

15. SECURITY CLASS. (of this report)
UNCLASSIFIED

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)
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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Six poly(fluoroalkoxy) phosphazene (PNF) elastomers were examined against a Viton control as candidates for O-rings which can function between -65° F and +350° F. The six ~~All~~ PNF-compounds were based on a single polymer manufactured by Firestone Tire and Rubber Company. Two compounds were purchased from Firestone and three from Horizons, Inc.; Nichols Engineering, Inc. supplied a proprietary compound. These six vulcanizates represent the most (over)

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S/N 0102-014-6601

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

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20. ABSTRACT (Cont'd fr p1473A)

advanced compound technology for PNF. Tensile data clearly show that these six PNF compounds cannot withstand hydraulic fluid MIL-H-83282A at +350° F. The tensile strengths deteriorate rapidly with immersion time. By contrast, the Viton control shows almost no change. Under The compression set characteristics of the six PNF compounds show a significant improvement over those previously examined, in previous work. Nevertheless, it is expected that compression set, too, would suffer with increased conditioning time at +350° F in the hydraulic fluid. The low temperature flexibility of PNF vulcanizates is excellent, but other important physical properties show obvious deficiencies, particularly after conditioning in hydraulic fluid at +350° F.

(Authors)

ADMINISTRATIVE INFORMATION

This work was accomplished under NAVAIR WF 54 544 205, Work Unit 2841-516, as outlined in reference (a). The technical coordinator is Mr. J. Gurtowski, NAVAIR (AIR 52032C); the project engineers are Dr. Paul Lagally and Messrs. W. Klemens and H. S. Preiser.

ADMINISTRATIVE REFERENCE

(a) NAVAIR Program Summary of 1 May 1974

LIST OF ABBREVIATIONS

FKM	- fluorocarbon rubber
hr/° F	- hour per degrees Fahrenheit
in-lbs/in^3	- inch-pound per cubic inch
min/° F	- minute per degrees Fahrenheit
ml	- milliliter
mm	- millimeter
NBR	- nitrile-butadiene rubber
PNF	- poly(fluoroalkoxy)phosphazene
psi	- pounds per square inch

TABLE OF CONTENTS

BACKGROUND

Nitrile-butadiene rubber (NBR) O-rings have had an acceptable service life in aircraft hydraulic systems which function between -65° F (-53.9° C) and +225° F (+107.2° C). However, service life is reduced and maintenance requirements are increased at temperatures which approach and exceed +300° F (+148.9° C) in the more advanced aircraft. Viton fluorocarbon rubber (FKM) O-rings function up to +450° F (+232° C) but lose their flexibility below -20° F (-28.9° C). Development is presently directed toward materials which can function as O-rings between -50° F (-45.6° C) and +350° F (+176.6° C).

Over the last several years, attention has focused on inorganic polyphosphazene elastomers which are stable over a wide temperature range. The Firestone Tire and Rubber Company manufactures pilot quantities of the organic substituted poly (fluoroalkoxy) phosphazene, trade name PNF. Earlier work[1] revealed that the physical properties of three compounds made from this polymer (two from Horizons, Inc., and one from Firestone) degraded severely when immersed at +350° F (+176.6° C) in the hydraulic fluid specified in MIL-H-83282 Amendment 2 (for service between -40° F (-40.0° C) and +400° F (+204.4° C)).

Hydraulic fluid MIL-H-83282A (for service between -50° F (-45.6° C) and +400° F (+204.4° C) is currently being introduced into military aircraft. The fluid has a synthetic hydrocarbon base and is fire-resistant.

OBJECTIVE

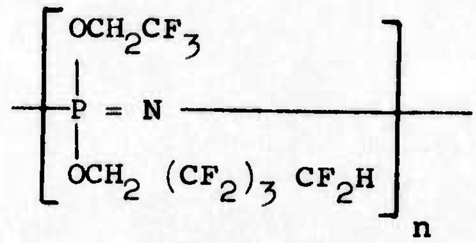
The objective of this project is to further examine compounded PNF vulcanizates as candidates for O-rings for service between -50° F (-45.6° C) and +350° F (+176.6° C) in hydraulic fluid MIL-H-83282A.

INVESTIGATION

MATERIALS

The six poly(fluoroalkoxy) phosphazene compounds examined are based on the same elastomer gum (PNF) which has the following general formula:

[1] Superscripts refer to similarly numbered entries in the Technical References at the end of the text.



Three of the compounds were purchased from Horizons, under Contract N61533-75-M-3055 and two from Firestone under Contract N61533-75-M-2950. The sixth compound, designated NE-P-18 was supplied by Nichols Engineering, Inc., Shelton, Connecticut. The formulation of this compound is proprietary to Nichols. These six compounds represent the most advanced compounding technology for PNF. The Viton reference control was compounded at DTNSRDC and was chosen for its known stability to hot hydrocarbon hydraulic fluids. Formulations for the compounds are included in table 1.

The hydraulic fluid used in the immersion conditioning meets specification MIL-H-83282A and was supplied by Royal Lubricants as batch F8801.

TABLE 1
COMPOUNDS FORMULATIONS

Ingredient	Supplier						
	DTNSRDC	Firestone		Horizons			Nichols
	Compound Identification						
	Viton 1296	R194887	R194888	2104-44	2104-45	2104-46	NE-P-18
Viton E-60-C	100	-	-	-	-	-	-
PNF	-	100	100	100	100	100	100
Quso WR82	30	30	30	25	10	25	*
Nulock 321L	-	-	-	10	40	10	*
Stan Mag ELC	-	6	6	-	-	-	*
Elastomag 170	-	-	-	-	6	-	*
Elastomag 20	-	-	-	5	-	5	*
Maglite D	3	-	-	-	-	-	*
Stabilizer $(8\text{-HQ})_2Zn$	-	2	2	-	-	-	*
Chemlink 30	-	-	-	1.5	1.5	-	*
Silane A-172	-	-	-	-	-	2	*
Teflon 6	-	-	6	-	-	-	*
Calcium Hydroxide	6	-	-	-	-	-	*
Lucidol 78	-	-	-	-	1.5	-	*
Luperco 101XL	-	-	-	1.5	-	1.2	*
Vulcup R	-	0.36	0.36	-	-	-	*
Press Cure min/° F	60/320	24/340	24/340	15/350	15/230	15/350	
Oven Cure hr/° F	24/450	-	-	0.5/350	0.5/350	0.5/350	

*Proprietary.

METHODS

Tensile properties were determined according to ASTM D 3196-73T. Values were calculated from the average cross sections and the average loads of five specimens. A forced draft oven was used for conditioning in hot hydraulic fluid. Fifty ml* of fluid covered 5 specimens suspended on a thin stainless steel wire in a 32 x 200 mm test tube. Specimens in the fluid were agitated daily.

Compression set was determined according to ASTM D395-69 Method B. Specimens consisted of stacked disks obtained when the tensile ring specimens were cut. The constant deflection rack was immersed in the hydraulic fluid and conditioned either in a forced draft oven or low temperature cold box.

The torsional stiffness of compounds was measured according to ASTM D1053-73. Iso-octane was the liquid medium for all temperatures except room temperature where air was the gaseous medium.

RESULTS AND DISCUSSION

Figure 1 clearly shows that the six PNF compounds cannot withstand continuous exposure to hydraulic fluid at +350° F (+176.6° C). Tensile strength deteriorates rapidly with immersion time. By contrast, the Viton reference control shows almost no change through the 14-day immersion period.

Data plotted on figure 2 shows that the six PNF compounds exhibit stability when heated for 14 days in the fluid at +300° F (+148.9° C), but continued immersion through 28 days shows a considerable loss in tensile strength. The Viton control shows little change through the 28 days immersion as observed previously. Although aircraft seals rarely experience high temperature in excess of two hours, the effects are cumulative over the life of the seals. Therefore, 28-day continuous immersion test is regarded as realistic.

Figure 3 illustrates the effect of increasing the immersion temperature from +300° F (+148.9° C) to +350° F (+176.6° C) on the strain energies of the six PNF compounds and the Viton control. The PNF vulcanizates deteriorated while the Viton was stable. Strain energy measurements are useful indicators of potential performance of materials under cyclic loading.

Ultimate elongations are generally stable through the 28-day immersion period at both +300° F (+148.9° C) and +350° F (+176.6° C) as shown in tables 2 and 3 on tensile properties.

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*A list of abbreviations used appears on page i.

TABLE 2
TENSILE PROPERTIES
AFTER CONDITIONING IN MIL-H-83282A AT +300° F

Tensile Properties (ASTM D3196-73T)	Immersion Time, days	Supplier: DTNSRDC	Firestone		Horizons			Nichols
		Compounds						
		Viton 1296	PNF 194887	PNF 194888	PNF 2104-44	PNF 2104-45	PNF 2104-46	PNF NE-P-18
Tensile strength, psi	0	1900	1540	1660	1550	1850	2010	1090
	3	1800	1240	1530	1310	1670	1560	910
	7	1480	-	1390	1040	1360	1510	930
	14	1490	1130	1330	1080	1290	1520	860
	28	1740	720	880	520	1050	810	320
% retained	28	92	47	53	34	57	40	29
Ultimate Elongation, %	0	140	120	110	120	90	120	120
	3	150	120	120	130	90	120	140
	7	160	120	130	140	90	140	160
	14	160	140	130	150	90	150	160
	28	180	150	120	120	80	150	80
% retained	28	128	125	109	100	89	125	67
Strain Energy, in-lbs/in^3	0	1610	680	900	780	710	940	660
	3	960	610	780	700	700	650	630
	7	1270	-	810	630	540	810	770
	14	1320	575	820	690	490	880	780
	28	1880	540	640	300	400	620	180
% retained	28	116	79	71	38	56	65	27
Hardness, Shore A, points (ASTM D2240-68)	0	77	50	60	63	62	58	70

TABLE 3
TENSILE PROPERTIES
AFTER CONDITIONING IN MIL-H-83282A AT +350° F

Tensile Properties (ASTM D3196-73T)	Immersion Time, days	Supplier: DTNSRDC	Firestone		Horizons			Nichols
		Compounds						
		Viton 1296	PNF 194887	PNF 194888	PNF 2104-44	PNF 2104-45	PNF 2104-46	PNF NE-P-18
Tensile strength, psi	0	1900	1540	1660	1550	1850	2010	1090
	3	1910	1310	1590	1070	1420	1530	930
	7	1810	1130	1280	650	1300	1070	600
	14	1800	540	790	310	860	560	190
% retained	14	95	35	48	20	47	28	17
Ultimate Elongation, %	0	140	120	110	120	90	120	120
	3	160	140	140	130	90	140	160
	7	160	130	120	120	80	150	130
	14	160	100	100	100	80	150	70
% retained	14	114	83	91	83	89	125	58
Strain Energy, in-lbs/in^3	0	1610	680	900	780	710	940	660
	3	1660	670	1000	540	510	770	770
	7	1640	600	760	310	430	660	490
	14	1560	240	490	150	290	400	100
% retained	14	96	35	54	19	40	42	15
Hardness, Shore A, points (ASTM D2240-68)	0	77	50	60	63	62	58	70

The six PNF compounds examined showed a significant improvement of the compression set over those examined in previous work,[1] see table 4. However, from the stress-strain data cited above, it is expected that compression set, too, would suffer with increased conditioning time at +350° F (+176.6° C) in the hydraulic fluid.

TABLE 4
COMPRESSION SET
(ASTM D 395-69 METHOD B)

Compound Identification	% Set After 7 Days Conditioning in MIL-H-83282A	
	at -50° F	at +350° F
DTNSRDC		
Viton 1296	26	52
Firestone		
R 194887	9	65
R 194888	19	69
Horizons		
2104-44	5	63
2104-45	6	69
2104-46	3	36
Nichols		
NE-P-18	19	79

Torsional stiffness data in figure 4 (items (a) and (b) show the superior low temperature flexibility of the PNF vulcanizates contrasted to the Viton compound.

Young's Modulus at room temperature was calculated from the torsional stiffness data and is shown in table 5. Viton 1296 has the highest modulus which is reflected in the Shore A hardness values shown in tables 2 and 3. The PNF compounds would exhibit higher moduli if they were compounded to a higher Shore A value. Generally, a Shore A value of 80 is considered about optimum for an O-ring in this application. The PNF materials were between 50 and 70 in Shore A hardness contrasted to a value of 77 for the Viton.

TABLE 5 - YOUNG'S MODULUS
AT ROOM TEMPERATURE (ASTM D 1053-73)

Supplier	Compound	Young's Modulus psi
DTNSRDC	Viton 1296	7470
Firestone	194887	480
	194888	568
Horizons	2104-44	989
	2104-45	396
	2104-46	342
Nichols	NE-P-18	945

CONCLUSION

Future work on the sole PNF polymer available is not warranted for aircraft seal applications operating under temperature extremes of -50° F(-45.6° C) to +350° F(+176.6° C).

RECOMMENDATION

Until improved phosphazene or other polymers are developed, it is recommended that future efforts for high performance aircraft seal applications be directed toward the improvement of fluorocarbon compounds for low-temperature performance.

TECHNICAL REFERENCE

1 - Lagally, P., and W. Klemens, Elastomers for Service as Seals for Engine Lubricants and Hydraulic Fluids, NSRDC Rept MAT-74-42 (Oct 1974)

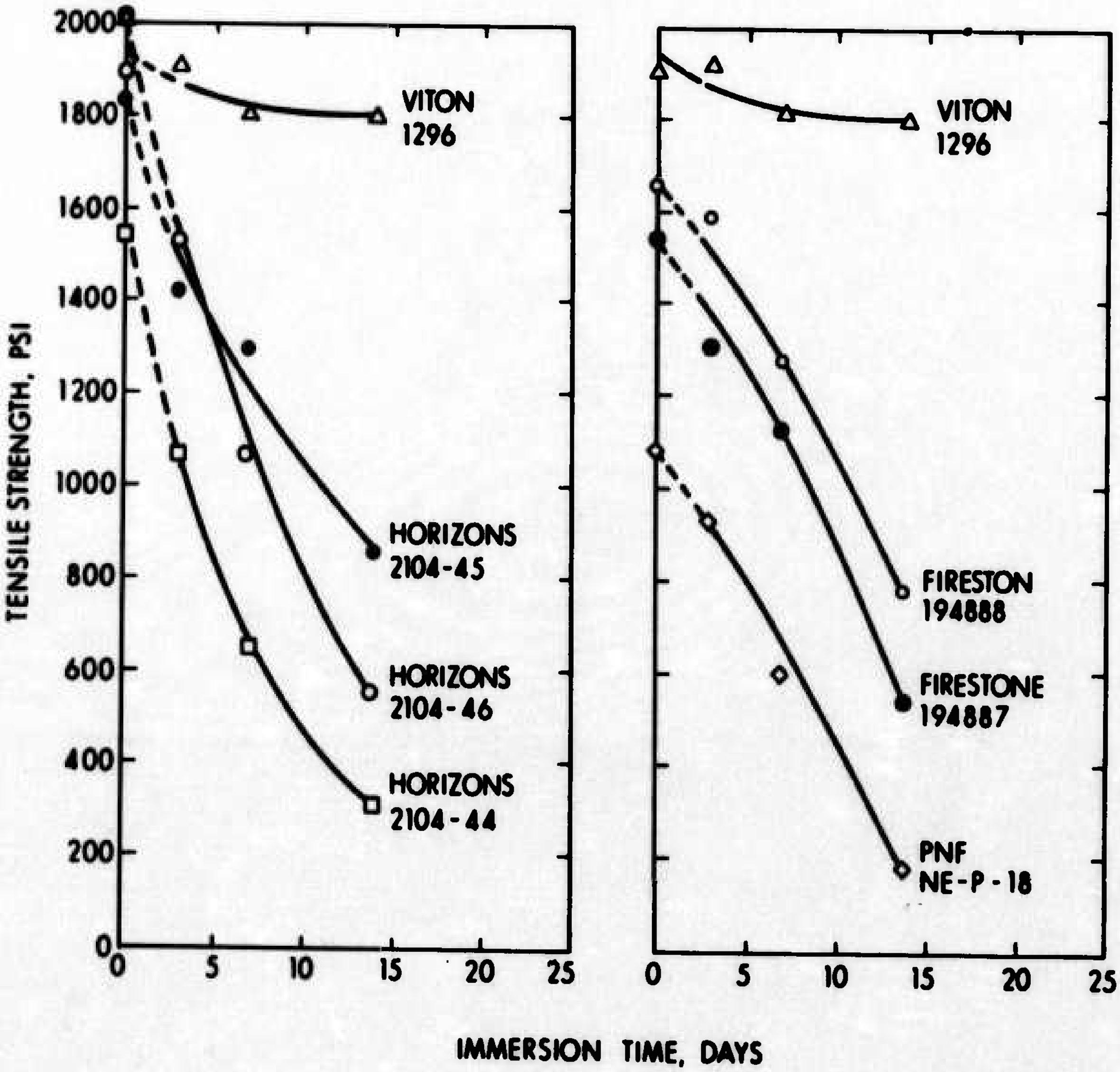


Figure 1 - Tensile Strength
After Conditioning in MIL-H-83282A at +350° F

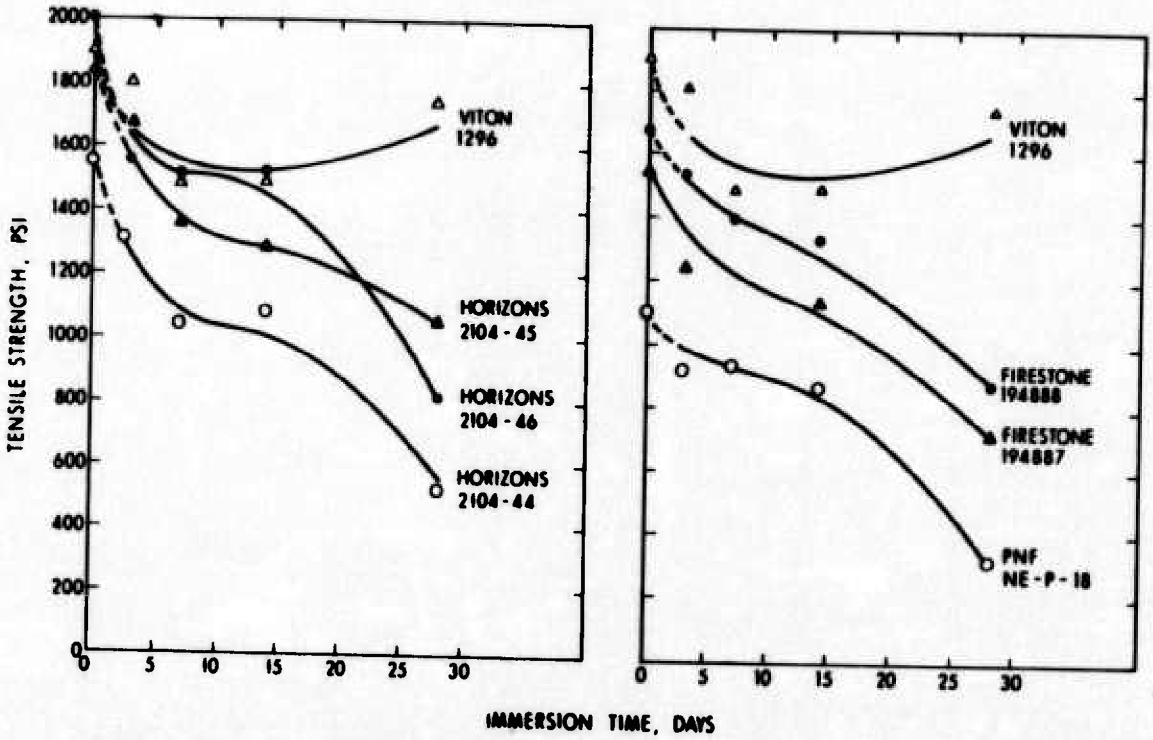


Figure 2 - Tensile Strength
After Conditioning in MIL-H-83282A at +300° F

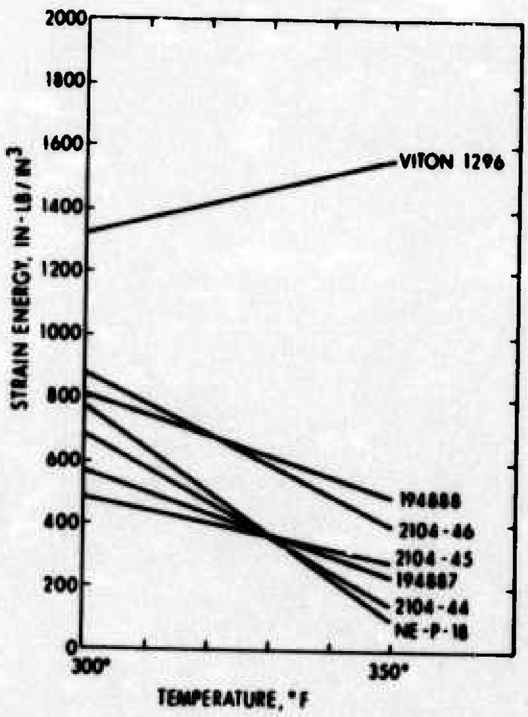
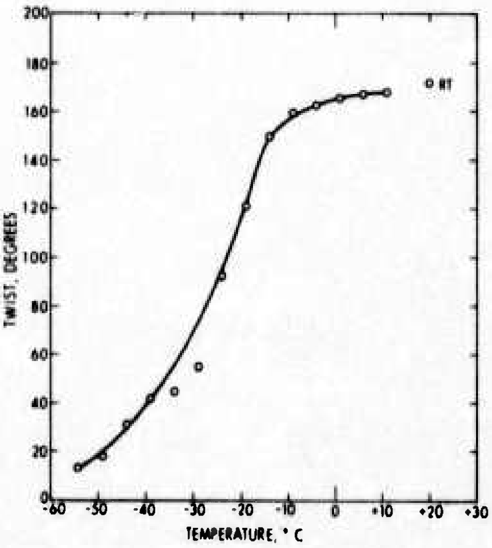


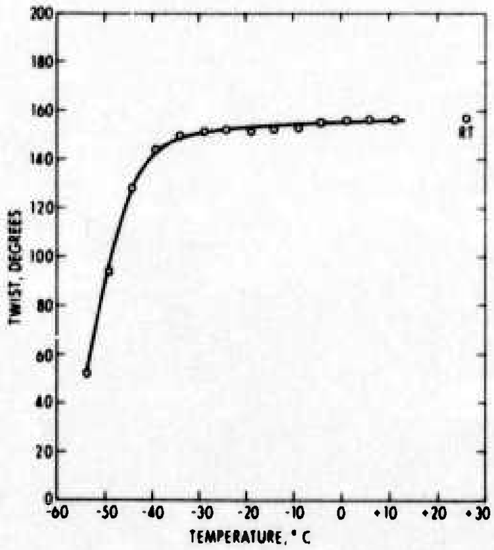
Figure 3
Strain Energy
Versus Temperature
14-Day Immersion
in MIL-H-83282A

Item (a)

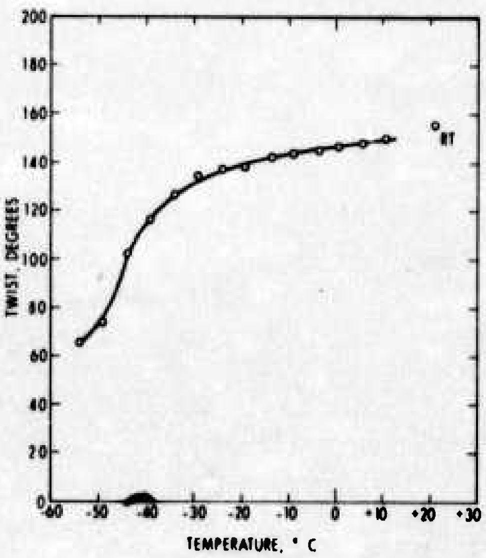
Viton 1296
White Wire



Firestone 194887
Black Wire



Firestone 194888
Black Wire



NE-P-18
Black Wire

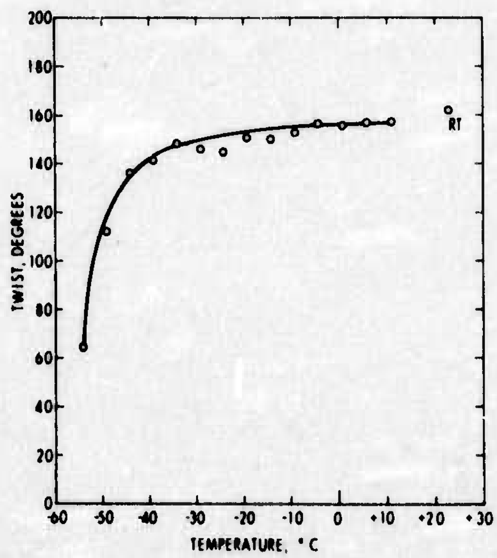
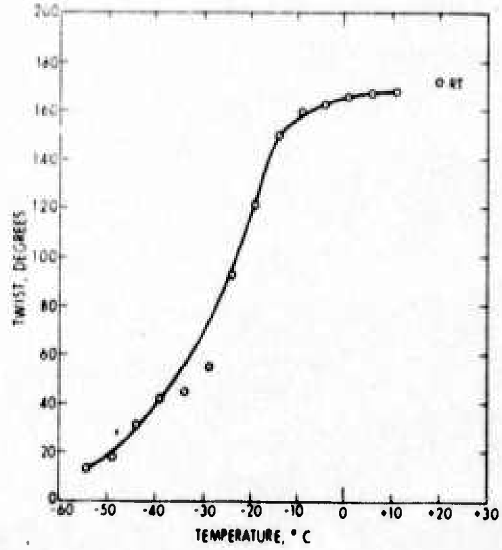


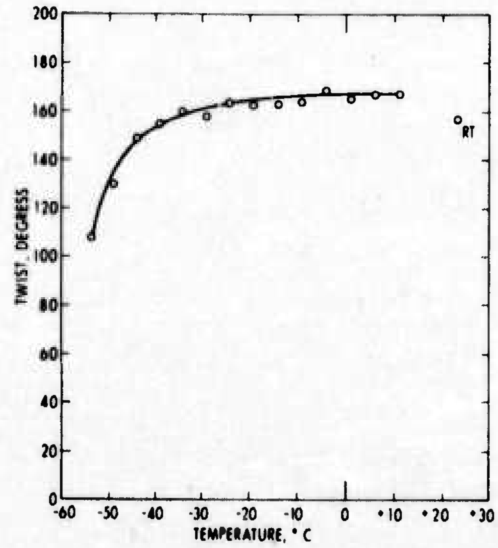
Figure 4
Torsional Stiffness (ASTM D 1053-73)

Item (b)

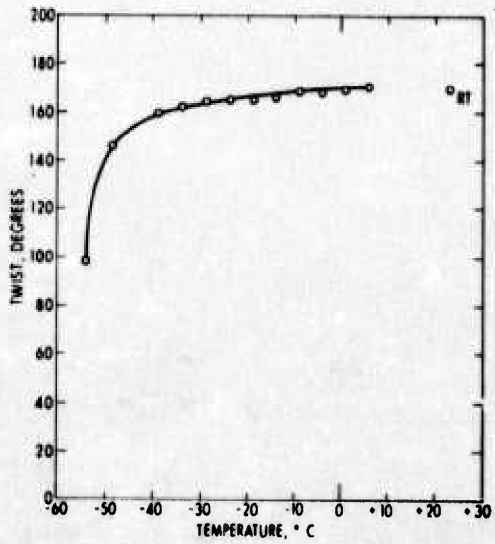
Viton 1296
White Wire



Horizons 2104-44
Black Wire



Horizons 2104-45
Black Wire



Horizons 2104-46
Black Wire

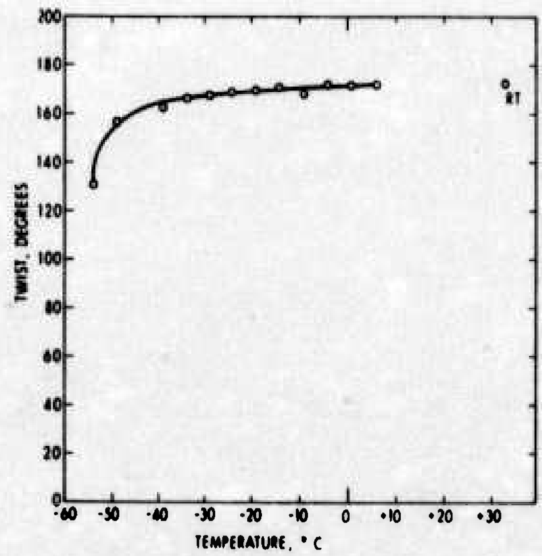


Figure 4 - Cont

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